# CHEMISTRY 2364 Fall 2009

INSTRUCTORS: Dr. Tamara Nauman (317 Williams; 578-5978)

Office Hours: MTWTh 8:30 am-9:30 am;

MW 2:30 pm-3:30 pm

Dr. Alfonso Davila (319 Williams; 578-9093)

Office Hours: MW 1:30-2:30 pm; TTh 2:30-3:30 pm

CHEM 1212 or 1431 and 2060, 2261 or 2461 and registration in 2262 or 2462 PRE-REQ:

**REQUIRED TEXT:** 

ORGANIC CHEMISTRY LABORATORY MANUAL, 3rd ed.

by Tamara Nauman (ISBN: 0-536-26019-2)

Organic Lab Lecture Notes Fall 2009 will be sold in lab by MSGSA for \$10. Outside of lab see Nick Gariano on TTh from 1-2 pm in 737 Choppin.

The Organic Chem Lab Survival Manual, 7th ed. HIGHLY RECOMMENDED:

by James W. Zubrick (ISBN: 978-0-470-12932-6)

SCHEDULE, Fall 2009: H = Handout; M = Manual									
AUG.	26-27	Check-in, Safety Lecture							
SEPT.	31-1 Decolorization and Separation by Extraction I (M 51-54)								
·	2-3	Separation by Extraction II (M 54-56) and Benzopinacol (M 81-83)							
	7-8	LABOR DAY HOLIDAY AND NO LAB DAY							
	9-10	TLC, Column Chromatography (M 57-59) and GC (H)							
	14-15	Organic Qualitative Analysis I (M 61-68); FT-IR (H)							
	16-17	Organic Qualitative Analysis II							
	21-22	Preparation of Cyclohexene (M 79-80)							
	23-24	Diels-Alder Reaction I (M 113-114); GC and FT-IR of Cyclohexene							
	28-29	MIDTERM REVIEW. Diels-Alder Reaction II (M 114-115), Pinacol (M 83-85)							
OCT.	30-1	NO LAB DAY AND FALL BREAK							
	5-6	TA Review and Make-up Lab							
	7	NO LAB DAY							
	8 (Thurs.)	MIDTERM EXAM [6:00-8:00 pm, Sec. 1, 3, 4, 5 in 103 Williams and							
	·	Sec. 6, 7, 8 in 102 Williams]							
	12-13	p-Nitroaniline I (M 87-92)							
	14-15	p-Nitroaniline II (M 92-93)							
	19-20	p-Nitroaniline III (M 93-94), Nylon [6,10] (M 99-100)							
	21-22	Fischer Esterification (M 95-96) and NMR							

26-27 Luminol (M 73-77)

Preparation of Adipic Acid (M 97-98) 28-29

Para Red Dye (M 105-107), Aldol (M 109-111) and Cannizzaro I (M 101-103) NOV. 2-3

Cannizzaro Reaction II (M 103) 4-5

Lidocaine I (M 117 -118) 9-10

Lidocaine II (M pgs. 118-119) 11-12

FINAL REVIEW; Make-up Lab 16-17 18-19 TA Review and Check Out

23 NO LAB DAY

FINAL EXAM [6:00-8:00 pm, Sec. 1, 3, 4, 5 in 103 Williams and 24 (Tues)

Sec. 6, 7, 8 in 102 Williams]

**GRADING:** Your final grade in the laboratory is based on your performance on graded work by the percentages that follow:

Notebooks (15%), Products (10%), Quizzes (15%), Midterm Exam (30%), Final Exam (30%)

Thus, your grade is not based on "how hard you worked in the lab" or for "your need to obtain a certain grade to keep a scholarship" or for "getting into med school or dental school". You receive the grade that you have earned.

# **GRADE ASSIGNMENTS:**

89.5 - 100 = A 79.5 - 89.4 = B 69.5 - 79.4 = C 59.5 - 69.4 = D Below 59.5 = F

#### LAB NOTEBOOK:

A carbonless copy notebook with consecutively numbered pages is required in this lab. You must write in ink and never use white out. Simply draw a line or cross through any mistakes. Do not plagiarize from the lab manual. You will receive a zero for plagiarized procedure and theory sections and you will be reported to the Dean. Your notebook grade is proportional to the time it takes your teaching assistant (TA) to grade it; therefore, the notebook should be well organized, thorough (but concise) and informative. A schedule of when laboratory notebook reports are due will be posted in the lab and on the CHEM 2364 bulletin board. Late reports will be penalized. TA's often request that you turn in the original and keep the carbon copy in the notebook because it is easier for them to read the original. At least, the first two pages in your notebook should be used for the "Table of Contents". Before you can perform an experiment, you will have written your prelab in your notebook. Pre-lab write up consists of Title, Purpose, Table of Chemicals and Procedure. An example of a Table of Chemicals entry follows:

Table of Chemicals								
Name	Formula/ Structure	Mol. Wt. (g/mol)	MP/BP (°C)	Density (g/mL)	Solubility	Hazards*		
ethanol	C <sub>2</sub> H <sub>5</sub> OH	46.07	-117.3/78.5	0.789	w, eth, ace, bz	flammable		

\*Hazards (chemical alerts) include toxic, corrosive, caustic, flammable, carcinogenic. This information as well as the other data can be found in the Middleton Library or on the internet. In the Library look in the <u>CRC Handbook of Chemistry and Physics</u>, the <u>Merck Index</u> or the <u>Aldrich</u> catalog. MSDS (Material Safety Data Sheets) and an <u>Aldrich</u> catalog are found in the organic labs.

For the **Procedure**, divide the page in half vertically. On the left hand side of the page, write the procedure in your own words and on the right hand side write the **Observations and Results** (see Example notebook entry below). The procedure should be concise but in enough detail that you could perform the experiment from your notebook without your lab manual. Sketch the apparatus if applicable.

Example notebook entry **Procedure**: Weigh out 23.5 g of NaCl.

Measure the MP of acetaminophen (lit. MP = 169-172°C).

**Observations and Results:** 

23.47 g of NaCl used. NaCl is a white, crystalline solid.

Melting range 168.5 - 173°C.

Observations and Results are written on the right hand side of the page as you perform the experiment (write directly in your notebook; do not write on scratch paper for later transcription.) Observations include color changes, bubbles, temperature changes, formation of precipitates, etc. Results are weight, physical data such as MP or BP, appearance of products, FT-IR or GC data and when performing synthesis, percent yield calculation (show all work). Next you write the **Theory**, **Discussion and Conclusion**.

For the Theory give a brief discussion of the chemistry in the experiment including chemical reactions and complete arrow-pushing mechanisms (unless otherwise advised). Discuss the techniques used, such as refluxation, recrystallization, distillation, separation, and the principles behind how they work. For the Discussion section, restate your final results, such as MP, BP, % yield, appearance of products, and compare them to literature values where appropriate. Discuss the results of the experiment thoroughly. Include sources of reduced yield such as spillage, failure to control temperature, etc. and offer ways that you could have improved your results. For the Conclusion, argue why you are certain that you made the desired product.

### PRODUCTS:

You must submit a sample vial with your TA's name, your name, your lab partners name and the name of the compound contained in the vial to your TA. Products are graded on a 10 point scale. If you submit an empty vial but performed the experiment, you will receive a minimum of 6 points. You are not allowed to work in combined teams to submit a product.

# **QUIZZES:**

A short 15 point quiz covering the previous lab will be given at the beginning of each lab. This serves as a way of making sure that you have learned the material and helps prepare you for the midterm and final exams. A schedule of quizzes will be posted in the lab and on the CHEM 2364 bulletin board. You will be allowed to take two make-up quizzes given on the dates of the make-up labs. These make-up quizzes will replace your lowest quiz at midterm and your lowest quiz after midterm. If you are late to class and miss the quiz, you will receive a zero for that quiz. No make-up quizzes will be given. There are only two make-up labs for those people who have a valid excuse (one before midterm and one at the final). If you miss more than 2 labs you will be given a ZERO for those additional missed labs. Only exceptions are University excused absences and in those cases the lab grade will be pro-rated if you are unable to complete them during the make-up lab. You will still be responsible for the material covered in these labs on the midterm and final exams. Make-up lab times are designated on the schedule. No bonus work will be given.

# **MIDTERM AND FINAL EXAMS:**

The midterm exam will be given on Thursday, Oct. 8 from 6-8 pm and the comprehensive final exam will be given on Tuesday, Nov. 24 from 6-8 pm. These exams are closed notebook and closed manual exams. At midsemester, your midterm exam is worth 60% of your overall grade and at the end of the semester the midterm is worth 30% and the final is worth 30% of your overall grade. Examples of old midterm and final exams including a few answer keys will be found on Moodle.

In studying for the midterm exam, you may want to look at the final exams because it is comprehensive. Also, we now do the Benzopinacol/Pinacol Rearrangement and the Diels-Alder reaction before midterm and these questions can be found on the old final exams. In studying for the final exam, you will need to look at Luminol questions on the old midterm exams. A detailed study guide will be handed out at least a week before the midterm and final exams.

# **INSTRUMENTATION ROOM:**

The Organic Lab Instrumentation Room is located in 315 Williams (across from the organic labs). The instruments that you will be using in this room are FT-IR and gas chromatographs (GC). Lab goggles are required in this room. Bring your product in a labeled vial and remember to take it back with you to your lab. Dispose of any used materials in the appropriate waste container before leaving.

#### SAFETY & HOUSEKEEPING:

The safety and housekeeping rules for the Organic Lab are found in the lab manual on pages 3 through 6 with changes and additions listed below. It is mandatory that you obey these rules each lab otherwise you will lose a letter grade on your notebook grade for the day of the infraction (repeated violations will result in you being dismissed from the lab). After three ejections you will be dropped from the lab course.

- Wear safety goggles (no safety glasses are allowed) at all times when lab drawers are opened and glassware is out on the benchtop.
- Empty traps.
- Remove everything from the sink troughs (hoses, paper, clamps, etc.).
- Cap reagent bottles immediately after use.
- Use a funnel when pouring organic liquid waste into the organic waste bottle and stopper the waste bottle with a cork stopper. DO NOT over fill waste bottle (there should be at least a 3 inch empty space between the liquid and the top of the bottle). DO NOT put in solutions with an aqueous layer. Separate the aqueous layer and neutralize if acidic before pouring down the drain with copious amounts of water and pour the water insoluble organic layer into the organic liquid waste bottle.
- Clean up spills immediately in the hoods, on the balances, on the floor, on the benchtops, etc.
- Return all common items such as pH paper, unused melting point capillaries, filter paper, etc. to the tray on the balance table. If any of these items are used up, go to the stockroom for replacements.
- DO NOT pipet from reagent bottles. Pour reagent into a labeled beaker and pipet from the beaker. Dispose of excess reagent at the end of the class in the appropriate manner.
- DO NOT put broken glass (including melting point capillaries) in the ordinary trash can. DO
  NOT put broken thermometer glass in the broken glass box (take it to the stockroom).
  Dispose of samples from vials in the appropriate waste containers and then discard the
  empty, clean glass vial in the broken glass box.
- DO NOT put gloves, cloth towels, paper, aluminum foil, glass vials, etc. in the solid chemical
  waste bucket in the hood. ONLY solid chemical waste is to be placed in this bucket. Scrape
  toxic chemical waste into the waste bucket in the hood and dispose of the weighing or filter
  paper in the trash can. Silica gel goes in the solid waste. Celite and decolorizing carbon go in
  the trash can not in the waste bucket.
- Turn the balances and the melting point apparati off at the end of the class.

#### **CHECK OUT:**

It is <u>mandatory</u> that you check out of the lab either Nov. 18 or 19 during your scheduled lab section. If you do not check out, then your final grade will be dropped by one letter grade.